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Document Number 5

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File: USPT

Oct 25, 1994

DOCUMENT-IDENTIFIER: US 5358765 A
TITLE: Cellulosic article containing an olefinic oxide polymer and method of manufacture

BSPR:

It is known that cellulosic articles may be made by a variety of procedures. For example, cellulose with or without chemical modifications, may be put into solution with a solvent, e.g. by dispersion or by dissolution, and then shaped into an article followed by solvent removal (with or without chemical modification of the cellulose) to solidify or set the shape of the formed cellulosic article. Examples of known processes for production of cellulosic articles are the viscose, cuprammonium, N-methyl-morpholine-n-oxide, zinc chloride and cellulose carbamate processes as described e.g. in U.S. Pat. Nos. 1,601,686; 2,651,582; 4,145,532; 4,426,228; 4,781,931; 4,789,006; 4,867,204; and 4,999,149, the teachings of which are all hereby incorporated by reference. The formed article may have a variety of shapes including a filament, bead, sheet or film. It is contemplated that the present invention may utilize any known method of producing a cellulosic article of any shape. With further respect to the present invention, although the article may be of any shape, preferred are films, generally having a thickness of 10 mils (254 microns) or less. Both planar, spherical, cylindrical and tubular articles are contemplated with seamless tubular articles being preferred and tubular films being especially preferred.

DEPR:

Surfactants suitable for use in the coating compositions on casings according to the present invention include those surfactants which act as wetting agents for the cellulosic casing surface and/or as emulsifying agents for oil such that the surfactant facilitates dispersion of the coating composition across the surface of the cellulosic casing. Nonlimiting examples of suitable surfactants include water dispersible or at least partially water-soluble surfactants such as alkylene oxide adducts of either fatty acids or partial fatty acid esters, for example, ethoxylated fatty acid partial esters of such polyols as anhydrosorbitols, glycerol, polyglycerol, pentaerythritol, and glucosides, as well as ethoxylated monodiglycerides, sorbitan trioleate, lecithin, and aliphatic polyoxyethylene ethers such as polyoxyethylene (23) lauryl ether.

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Document Number 2

Entry 1 of 9

File: USPT

Sep 14, 1999

DOCUMENT-IDENTIFIER: US 5951933 A

TITLE: Processes for precipitating tertiary amine oxide cellulose solutions containing water soluble polymers and products made therefrom

BSPR:

The use of organic N-oxides such as tertiary amine oxides for dissolving cellulose was first reported by Graenacher and Sallman in U.S. Pat. No. 2,179,181. Subsequently, the specific use of N-Methyl Morpholine-N-Oxide (NMMO) to dissolve cellulose was disclosed by D. L. Johnson in U.S. Pat. No. 3,447,939 and U.S. Pat. No. 3,508,941. These patents disclose the use of NMMO to dissolve cellulose and the production of films and fibers by the precipitation of the dissolved cellulose.

BSPR:

The use of NMMO as a solvent for cellulose and the production of cellulosic fibers and films was also disclosed in McCorsley et al., U.S. Pat. No. 4,142,913 which disclose a process wherein cellulose is mixed with a tertiary amine oxide such as NMMO and a liquid non-solvent containing controlled amounts of water which assists in intimately associating the tertiary amine oxide with the fibers of the cellulose to facilitate absorption of the tertiary amine oxide. The resulting mixture is maintained at a temperature at which the non-solvent and excess water are removed so that the cellulose dissolves in the tertiary amine oxide until a solution is obtained which is suitable for shaping into a cellulosic article such as by spinning or extrusion. The non-solvent can be water or it can be a mixture of water and organic non-solvent with a boiling point below 130.degree. C. including alcohols such as n-propyl alcohol, isopropyl alcohol, butanol or an aprotic liquid such as toluene, morpholine, methyl ethyl ketone or tetrahydrofuran.

BSPR:

McCrosley et al., U.S. Pat. No. 4,144,080 disclose a process wherein a comminuted solid precursor of a solution of cellulose in amine oxide such as NMMO is charged to an extrusion apparatus, is heated to a temperature where the amine oxide dissolves the cellulose to form an extrudable solution of cellulose and the resulting solution is extruded through a die to form an extrudate of uniform composition. Franks et al., U.S. Pat. No. 4,145,532 disclose methods of dissolving cellulose in solutions containing water and NMMO. Turbak et al., Chemtech, p. 51-57, January, 1980 provide a review of developments in cellulose solvent systems including amine oxides. Turbak subsequently reported cellulose solutions with lithium chloride and dimethylacetamide. These references further describe the potential use of such solutions in the production of new fiber and film products.

BSPR:

The NMMO process for producing cellulosic fibers and films has become particularly attractive in recent years because of safety and

environmental concerns regarding the viscose process traditionally used for production of cellulosic films and fibers. In particular, the use of carbon disulfide in the viscose system has led to a desire for a simple, more ecologically friendly closed loop totally recoverable cellulose solvent system. Use of processes for spinning cellulose from NMMO solutions continues with the manufacture of over 120 million lbs/year of cellulose fibers via the NMMO process in about 1996.

BSPR:

While the NMMO system for production of cellulosic films and fibers provides various benefits over use of the viscose system it is also subject to certain limitations. This is particularly the case with respect to the ability to control the precipitation of the cellulose. In the viscose system, the cellulose is first made into a xanthate derivative by the use of caustic soda and carbon disulfide. This derivative is then spun into a coagulation/regeneration bath containing high salt and low to medium acid so that the cellulose can first be congealed into a gelatinous mass and densified by the salt in a controlled manner. This allows the xanthate solubilizing groups on the cellulose molecules sufficient opportunity to permit alignment and packing of the cellulose into the proper positions to make a good quality product. The longer the regeneration is retarded, the higher is the resulting product quality since more effective stretching and alignment can be obtained in the densifying coagulated system.

BSPR:

In contrast to methods of using the viscose system, the cellulose molecules in tertiary amine oxide processes are not derivatized but are directly dissolved by the action of the tertiary amine oxide. This dissolving takes place over very narrow limits of water content. As may be seen from the graphs published by Franks et al. in U.S. Pat. No. 4,145,532 and also by Chanzy et al. "Swelling and Dissolution of Cellulose in Amine Oxide/Water Systems," Ninth Cellulose Conference, State University of New York, Syracuse, N.Y., May 24-27, 1982) there are relatively narrow concentration ranges for dissolving and maintaining cellulose in solution. Beyond these ranges, for example, further addition of water causes very rapid and drastic precipitation of the cellulose out of solution. See also, Turbak, TAPPI Journal, Vol. 67., No. 1 pp. 94-96 (1984). Thus, as compared to the viscose process, the ability to control the coagulation and precipitation of cellulose in a tertiary amine oxide process is substantially diminished thus hindering the ability to provide for orderly spinning of a fiber or extrusion of a film. While such rapid and dramatic precipitation is advantageous from the view of getting high spinning speeds, it is disadvantageous with respect to being able to control and improve the nature of the product properties. Accordingly, there exists a need in the art for methods to retard and control the rapid precipitation of the cellulose from NMMO and other tertiary amine oxide solutions in order that better control of molecular structure can be obtained during the coagulation and precipitation of the cellulose molecules.

BSPR:

The solutions may optionally incorporate di- tri and multiple esters as described in co-owned and copending U.S. Ser. No. 08/899,425 filed simultaneously herewith, wherein the disclosure is incorporated herein by reference. Preferred esters include glycerol diesters, and glycerol triesters including glycerol trioleate, glycerol monooleate diacetate, glycerol triacetate, as well as ethylene and propylene glycol fatty acid esters, lecithin, and citric acid esters. Also incorporated by reference herein is the disclosure of co-owned and copending U.S. Ser. No. 08/899,538 filed simultaneously herewith which describes improved methods for retarding and controlling the rapid precipitation of cellulose from tertiary amine oxide solutions in aqueous hardening baths by reducing the concentration of water therein and preferably increasing the concentration of tertiary amine oxides in the stead of the water therein.

DEPR:

According to this example, an approximately 10% solution of cellulose is prepared in N-methyl morpholine-N-oxide (NMMO) according to the general methods described in U.S. Pat. Nos. 3,447,939; 4,145,532; 4,426,288; 4,142,913; 4,144,080 and 4,145,532. The cellulose is first premixed with the desired additive before the addition of the NMMO and the subsequent removal of excess water to form the cellulose solution. In a typical run, 10 parts of cellulose is added to 80 parts of water containing 2 parts of dissolved sodium carboxymethyl cellulose (CMC), a water soluble polymer of 50,000 molecular weight. Since the coagulating water must be concentrated and recycled to save the NMMO, one of the reasons for using water soluble polymers having a molecular weight of more than 10,000 is to be sure that none of the relatively water soluble polymer will dissolve out of the film and contaminate the coagulating water. The system is mixed thoroughly to allow good intermixing of cellulose and CMC. To this mixture is then added 76 parts of NMMO and the mixture is placed in a sigma blade high torque mixer under vacuum. The mixing system is heated up to no more than 120.degree. C. while vacuum is continued and excess water is removed. (Safety note: 120.degree. C. is chosen since NMMO is known to explode violently at or above 140.degree. C.). When the active water level in the mixture reaches less than about 27% of the weight of the NMMO, preferably from 8%-15% the weight of the NMMO, both the cellulose and CMC are in solution. At the 15% weight of NMMO water level, this gives a solution containing essentially 10% by weight of cellulose, 2% by weight of CMC dissolved in 76.4% of NMMO containing 11.5% of water. This solution containing about 20% by weight of CMC based on the cellulose is then pumped to a screw feeder extruder, deaerated and extruded into the desired shape into an aqueous system precipitation bath. The precipitation bath can be cold or warm water or even steam. Regardless of the nature of the precipitation aqueous system, the rate of precipitation of the cellulose is significantly retarded and the extrudate can be subjected to more stretch and congealing than is possible if the additive is absent.

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Document Number 8

Entry 9 of 9

File: USPT

Jan 6, 1976

DOCUMENT-IDENTIFIER: US 3931027 A

TITLE: Cellulose material treated with a thermosetting resin and having improved physical properties at elevated temperatures

DEPR:

In accordance with the invention, the cellulose material is treated with an aqueous dispersion of a liquid, uncured, thermosetting resin and an amine curing or crosslinking agent. The resin can take the form of an epoxy resin, either glycidyl ethers or esters, with the preferred epoxy resin being the diglycidyl ether of bisphenol-A and epichlorohydrin. In addition, dispersible, aromatic diclycidyl esters and cycloaliphatic diglycidyl esters can also be utilized as the epoxy resin component. The thermosetting resin can also be a polyester resin which is a condensation polymer resulting from the reaction of dicarboxylic acids and polyhydric alcohols, as for example, when maleic acid or anhydride is reacted with glycerol, ethylene glycol, diethylene glycol, sorbitol, mannitol, polyethylene glycol and the like. To aid in dispersing the resin in the aqueous medium, the resin may contain a conventional dispersing agent.

DEPR:

A protein material, such as casein, isolated soy protein, or collagen type protein can be incorporated in the treating liquid in an amount of 0.1% to 10% by weight of the cellulose material, based on 100% solids. The protein material includes amine groups which aid in the crosslinking reaction and also contribute thermal stability to the treated cellulose. As a further advantage, the protein material acts as a film-former and serves to retain the resin in suspension or dispersion in the aqueous liquid. To solubilize the protein material in the aqueous solution, a small amount of a water soluble, highly basic amine, such as morpholine or diglycolamine, can be added to the solution. The basic amine is generally used in an amount of 0.1 to 5.0% of the cellulose fiber material to be subsequently treated. The protein material is soluble in the highly basic amine, which in turn is miscible with water, thereby enabling the protein material to be solubilized in the aqueous treating medium.

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Document Number 3

Entry 5 of 9

File: USPT

Aug 30, 1994

DOCUMENT- IDENTIFIER: US 5342872 A

TITLE: Peelable and recoverable aqueous film-forming composition

BSPR:

Another peelable or strippable film is disclosed in Martenson, U.S. Pat. No, 4,792,464. This film is a temporary, readily strippable, protective film for metal surfaces which incorporates corrosion inhibitors and is impervious to gases such as oxygen and water vapor. The film is applied as an aqueous composition comprising (1) a terpolymer of polyvinyl butyryl, polyvinyl alcohol, and polyvinyl acetate; (2) a hydroxyalkyl cellulose ester; (3) a corrosion inhibitor dispersed in a plasticizer for the polyvinyl butyryl; and (4) morpholine as a leveling agent. This composition is apparently designed simply as temporary, protective coating for metals, and the reference apparently does not contemplate use of the film as a painting mask. In any event, the reference fails to recognize the waste problem of requiring the recovery and reuse of the film.

DEPR:

The thickening agents employed in the aqueous film-forming composition may be useful to increase the cohesion of the liquid composition during the drying process, as a suspending agent or protective colloid to improve the stability of the aqueous emulsion, and to improve the film-forming capabilities of the composition. A representative, non-limiting list of useful water emulsifiable thickening agents includes carboxyl alkyl polymers such as carboxyvinyl polymers or carboxyl polymethylene; and starches such as potato starch. Additionally thickeners including water soluble polysaccharide materials such as xanthan gum are also useful in the composition of the present invention. To obtain multiple benefits, a combination of (1) a carboxyl vinyl polymer or potato starch and (2) xanthan gum is used as a thickening agent.

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Document Number 1

Entry 4 of 9

File: USPT

Sep 3, 1996

DOCUMENT-IDENTIFIER: US H001592 H
TITLE: Cellulosic food casing

BSPR:

An alternate cellulose production method involves forming a cellulose solution by means of a simple dissolution rather than requiring prior derivatization to form a soluble substance. A cellulose dissolution process is described in U.S. Pat. No. 2,179,181. This patent discloses the dissolution of natural cellulose by a tertiary amine oxide to produce solutions of relatively low solids content, for example, 7 to 10% by weight cellulose dissolved in 93 to 90% by weight of the tertiary amine. The cellulose in the resulting solution is nonderivatized prior to dissolution. U.S. Pat. No. 3,447,939 discloses use of N-methyl-morpholine-N-oxide (NMMO) as the cyclic amine solvent where the resulting solutions, while having a low solids content, can be used in chemical reactions involving the dissolved compound or to precipitate the cellulose to form a film or filament.

BSPR:

In particular, the present invention is a cellulose food casing comprising a sulfur free seamless extruded tubular film of a nonderivatized cellulose precipitated from a cyclic amine oxide solution of cellulose dissolved in N-methylmorpholine-N-oxide, which can be said to exhibit thermoplastic-like properties, and where the tubular film contains a water soluble softener and an additive such as a peeling aid, a coloring agent, and a flavoring agent.

DEPR:

Surfactants suitable for use in the coating compositions on casings according to the present invention include those surfactants which act as wetting agents for the cellulosic casing surface and/or as emulsifying agents for oil such that the surfactant facilitates dispersion of the coating composition across the surface of the cellulosic casing. Examples of suitable surfactants include water dispersible or at least partially water-soluble surfactants such as alkylene oxide adducts of either fatty acids or partial fatty acid esters, ethoxylated fatty acid partial esters of such polyols as anhydrosorbitols, glycerol, polyglycerol, pentaerythritol, and glucosides, as well as ethoxylated monoglycerides, sorbitan trioleate, lecithin and aliphatic polyoxyethylene ethers such as polyoxyethylene (23) lauryl ether.

DEPR:

It was not expected that the casing made from nonderivatized cellulose could be colored in the same fashion. As the solution prior to extrusion contains a large amount of NMMO in a small amount of water, about 78% NMMO in water, it is not an aqueous solution. It is believed that the addition of the aqueous color dispersion in the pre-extrusion stream would cause precipitation of cellulose, thereby blocking the extrusion of the film or casing.

DEPR:

139.5 grams of 784 NMMO/H.sub.2 O and 69.5 grams of a dope containing 154 cellulose and about 80% NMMO and 5% H.sub.2 O were combined. Next, 2.3 grams of a glycerin mixture of phthalocyanine blue pigment at about a 44 pigment level was added at 220.degree. F. to the dope mixture and mixed. This was cast into a film and regenerated with cold water, giving a blue film with even color throughout, made from pigmented nonderivatized cellulose.

CLPR:

1. A cellulosic food casing comprising a sulfur free seamless tubular film of nonderivatized cellulose precipitated from an amine oxide cellulose solution of cellulose dissolved in N-methylmorpholine-N-oxide (NMMO), wherein said casing is coated with an additive selected from the group consisting of a peeling aid, a coloring agent, and a flavoring agent.

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